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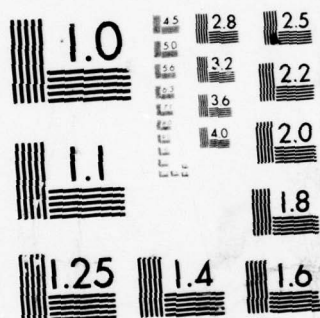
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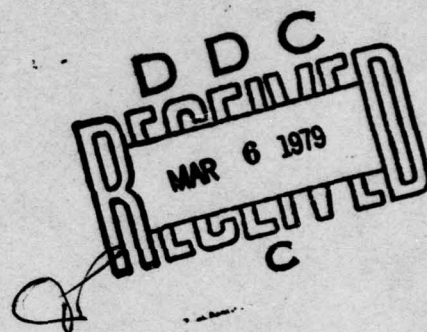
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CHEMICAL RELEASE PAYLOADS FOR
STRATOSPHERIC WIND MEASUREMENTS
1977-1978 AND RELATED PROGRAMS

Charles S. Stokes
William J. Murphy
Edward W. Smith

Germantown Laboratories, Inc.
4150 Henry Avenue
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30 October 1978

Final Report for Period - 4 January 1977 to 30 September 1978

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PREFACE

This report covers work performed on four series of chemical payload launches on sounding rockets. These launches took place at White Sands Missile Range, (WSMR), New Mexico. NASA Wallops Station, Virginia and Churchill Research Range (CRR), Manitoba, Canada.

These operations included design, fabrication, assembly and launch services for chemical payloads containing trimethyl aluminum (TMA) or titanium tetrachloride (TiCl_4) - methanol/water.

TABLE OF CONTENTS

	<u>PAGE</u>
A. Program and Description of Experiment	1
B. Payloads	1
C. Launch Operations	1
D. April 1977 Series White Sands Missile Range	2
E. May 1978 Series Wallops Island	2
F. September 1978 Series Fort Churchill, Canada	2
G. September 1977 Trimethyl Aluminum Launch White Sands Missile Range	3
H. Chemical and Physical Properties	3
I. Programmer Design	3
References	4
Appendix	21

A. Program and Description of Experiment

The program consisted of the local measurement of the turbulent transport properties of the stratosphere by use of a smoke trail produced by the simultaneous release of trimethyl aluminum and a 1:1 mole ratio of methanol and water from a Nike-Nike rocket vehicle or the release of trimethyl aluminum from a Nike-Tomahawk vehicle.

A total of eight payloads were flown. Seven utilized the titanium tetrachloride-methanol water system, one utilized the trimethyl aluminum system.

B. Payloads

The titanium tetrachloride payloads were similar to a type previously flown (1). The payloads consisted of three sections. The forward section consisted of a small aluminum nose tip and conical pressure vessel. The next section contained the programmer, batteries, explosive valves and appropriate liquid and gas plumbing. The third section was two cylindrical tanks in an annular arrangement. A Nike adaptor ring fitted the payload to the vehicle. Two types of this payload were used, one with a launch weight of approximately 550 pounds, the other with a launch weight of 685 pounds. Figures 1 and 2 show the general outline of these payloads, Figure 3 shows the flow diagram. (2, 3, 4, 5)

C. Launch Operations

The payloads were furnished by the Government and received by Germantown Laboratory unassembled. Each payload was completely assembled and checked, then disassembled for shipping. The Wallops Island launches were an exception, the payloads were assembled, checked, filled, and shipped by private truck. The programmer and explosive valves were installed at Wallops Island.

On arrival at the launch site (White Sands Missile Range or Churchill Research Range), the payloads were reassembled and checked. Whenever practical, filling operations took place out of doors or with an exhaust line leading outside because of the smoke problem with titanium tetrachloride. The alcohol-water system presented no problem in filling. After the programmer was checked and installed, the payload was taken to the launch area and mated to the Nike. The payload was pressurized on the launch rail as part of the flight checkout procedures.

Programmer development is covered under Section I. of this report.

The trimethyl aluminum payload was shipped filled to White Sands Missile Range by special ICC permit. The activator for the explosive valve was shipped separately. The programmer and activator were installed at White Sands Missile Range after checkout.

D. April 1977 Series White Sands Missile Range

Three payloads similar to a type previously flown were used in these experiments. Payload parameters are shown in Table I. Programmer timing and trail release are shown in Table II. Figure 1 is a schematic of the Nike-Nike smoke system Mark II. Figure 2 is a flow diagram of the payload and Figure 3 is a schematic of programmer type 12-309. All payloads operated satisfactorily.

E. May 1978 Series Wallops Island

Two payloads similar to the April 1977 Series were used in the experiments. Payload parameters are shown in Table III. Programmer timing and trail release are shown in Table II. Refer to Figures 1 and 2 for the smoke system schematic and the flow diagram. Figures 4 and 5 show the programmer and timer schematics used in these payloads. All payloads operated satisfactorily, although payload #029 started 20 seconds later than it should have because of mechanical slippage in the timer cam.

F. September 1978 Series Fort Churchill, Canada

Three payloads similar to the May 1978 Series, but larger, were used in these experiments. Payload parameters are shown in Table IV, programmer timing and trail release are shown in Table V. Figure 6 is the payload schematic, Figure 7 is the flow diagram. Programmer type 12-309A is shown in Figure 8. This was the first time the Mark III smoke payloads were flown. Both operated satisfactorily.

G. September 1977 Trimethyl Aluminum Launch White Sands Missile Range

A single trimethyl aluminum payload was launched from White Sands Missile Range on September 24, 1977. It did not perform satisfactorily. Instead of a series of puffs followed by a trail, the valve remained open on the first puff, venting the entire contents of the tank and creating a very large single white cloud. It was later found that this failure was due to a very subtle anomaly in the programmer timer.

Figure 9 is a schematic of the payload, flow system and timer settings. Figure 10 is a schematic of the programmer type 52-1363.

H. Chemical and Physical Properties

Appendix 1 gives chemical and physical properties of payload chemicals used, titanium tetrachloride, methanol-water and trimethyl aluminum. The information for safety and handling of trimethyl aluminum is given in an Ethyl Corporation brochure. (6)

I. Programmer Design

Due to the difficulty encountered with the trimethyl aluminum flight, a new programmer was designed and flight tested on the May 1978 Series from Wallops Island. See Figures 4 and 5 for the programmer and timer schematics used for these flights.

REFERENCES

- (1) Anon., AFCRL Dynamics Experiment for the Winter Anomaly Program, Wallops Island, VA., January 1976.
- (2) Stokes, C.S., Murphy, W.J. and Smith, E.W., Experimental and Flight Evaluation of the Titanium Tetrachloride-Methanol/Water System for the Production of Smoke Trails, Final Report, Germantown Laboratories, Inc., AFCRL-TR-74-0496, June 30, 1974.
- (3) Vickery, W.K., Techniques for Depositing Visible Smoke Trails in the Stratosphere for Measurement of Winds and Turbulence, AFCRL-TR-0221, April 21, 1975.
- (4) Stokes, C.S., Murphy, W.J., and Smith, E.W., Chemical Release Payloads for Operation Post Aladdin 74, Operation Aeolus and Operation Harses (Smoke II), Final Report, Germantown Laboratories, Inc., AFCRL-TR-75-0625, August 31, 1975.
- (5) Stokes, C.S., Murphy, W.J., and Smith, E.W., Chemical Release Payloads for the Winter Anomaly Program (1976), Ice Cap Program (1976) and Operation Harses (1976), Final Report, Germantown Laboratories, Inc., AFGL-TR-76-0312, October 30, 1976.
- (6) Aluminum Alkyls...and Other Selected Metal Alkyls, Ethyl Corp., Revised April 1965.

TABLE I
WHITE SANDS MISSILE RANGE
PAYLOAD PARAMETERS, NIKE-NIKE SMOKE SYSTEM

Payload No.	025	026	027
Vehicle	NIKE-NIKE	NIKE-NIKE	NIKE-NIKE
CH ₃ OH/H ₂ O Aft Tank			
Wt. Chem. (lbs.)	118	118	118
Orifice (in.)	0.335	0.335	0.335
TiCl ₄ Fwd. Tank			
Wt. Chem. (lbs.)	135	135	135
Orifice (in.)	0.303	0.303	0.303
Nose Cone			
Pressure (PSIA)	100	100	100
Total Payload Wt. (lbs.)	548½	549	540
CG, in. From Nike Joint	25	25¼	24-3/4
Launch date	April 22, 1977	April 26, 1977	May 2, 1977

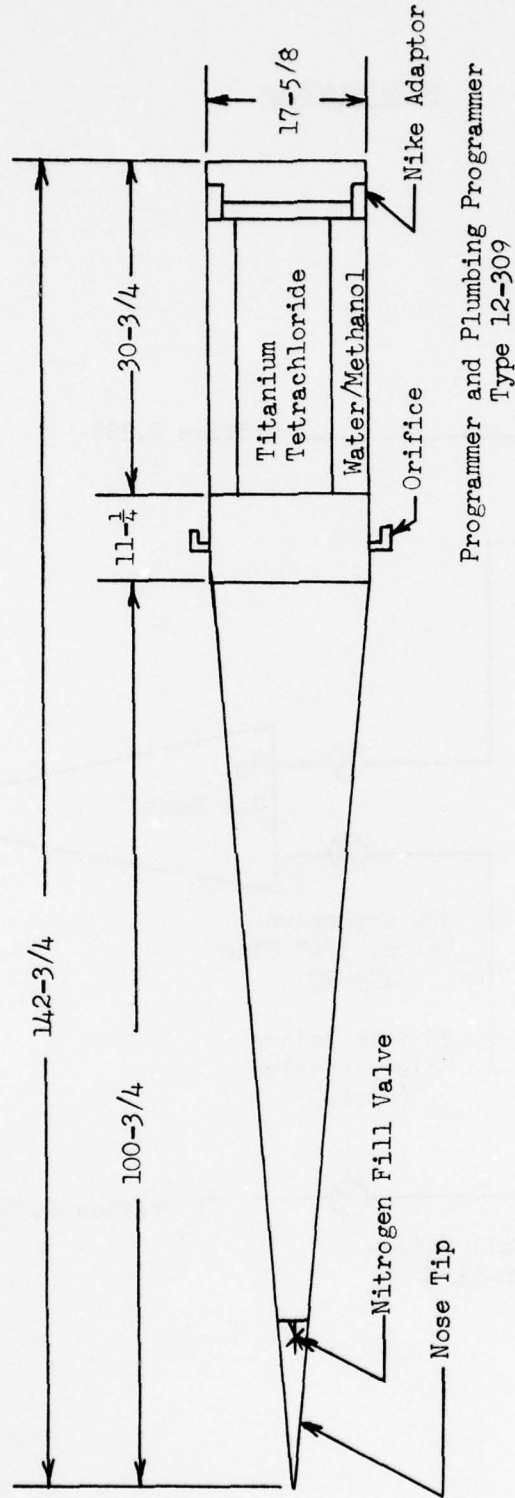
TABLE II
PROGRAMMER TIMING - TRAIL RELEASE
NIKE--NIKE SMOKE SYSTEM MARK II

Titanium Tetrachloride Orifice	0.303 inch
Tank Pressure	100 psia
Flow Rate	2.28 lbs./sec.

Methanol/Water Orifice	0.335 inch
Tank Pressure	100 psia
Flow Rate	2.00 lbs./sec.

<u>Trail No.</u>	<u>Start Second</u>	<u>End Second</u>
1	30	36
2	40	46
3	50	56
4	60	66
5	70	76
6	80	86
7	90	96
8	105	120

Programmer Type 12-309



PAYLOAD WEIGHT: 548 POUNDS

PAYLOAD CHEMICALS:

TITANIUM TETRACHLORIDE 135 POUNDS
 36 WT. % WATER/64 WT. % METHANOL 118 POUNDS

FIGURE 1

NIKE-NIKE SMOKE TRAIL PAYLOAD

FLOW DIAGRAM

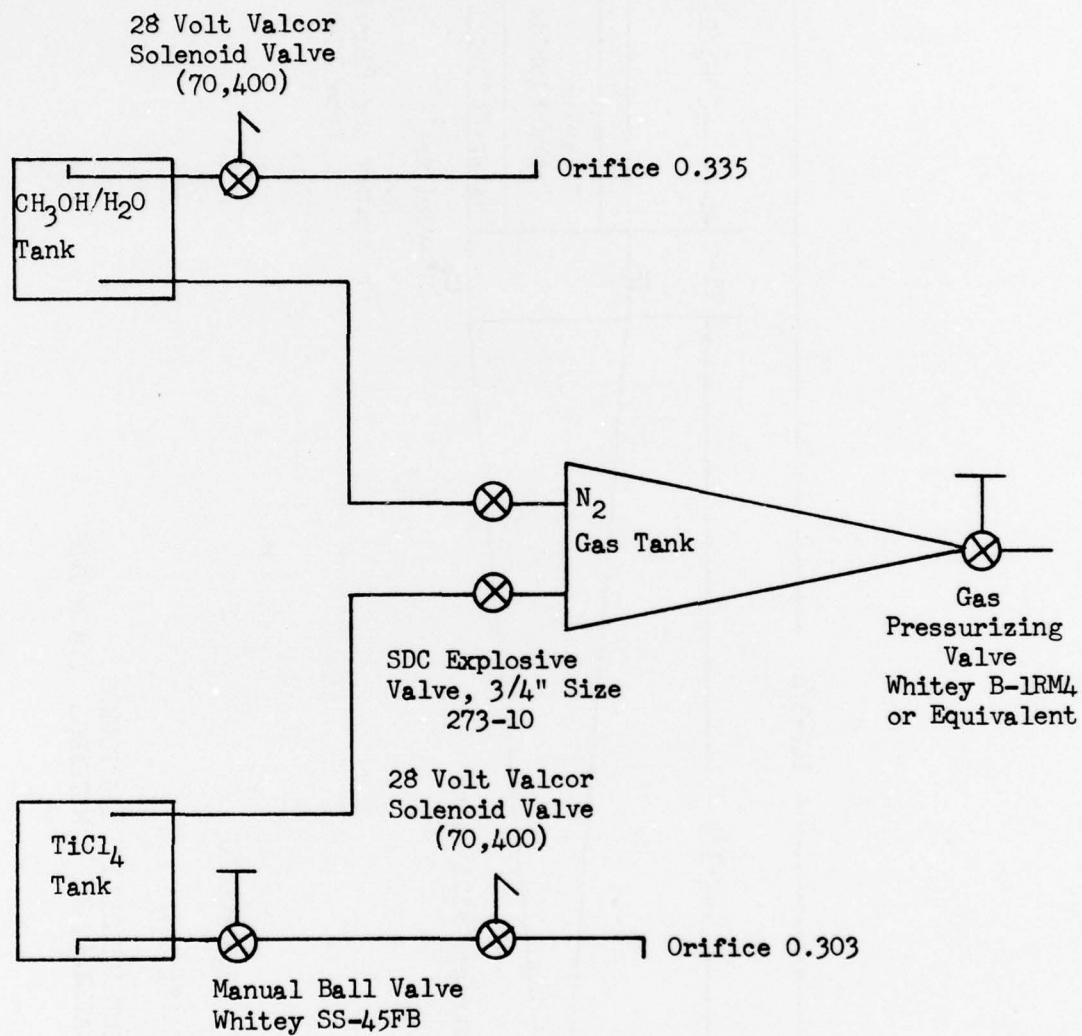
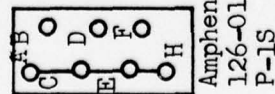
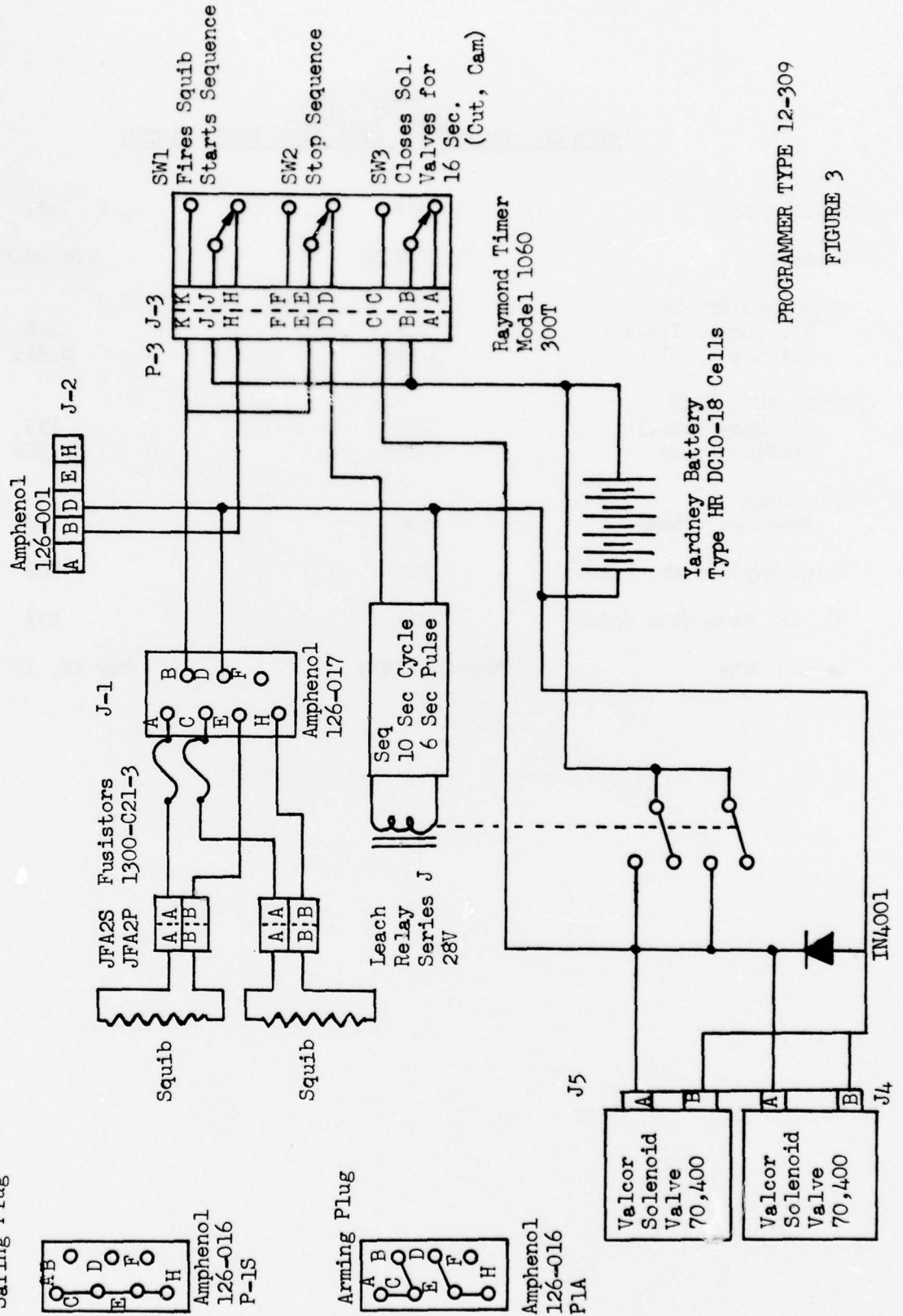
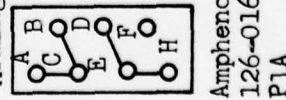


FIGURE 2

Safing Plug



Arming Plug



PROGRAMMER TYPE 12-309

FIGURE 3

TABLE III
WALLOPS ISLAND
PAYLOAD PARAMETERS, NIKE-NIKE SMOKE SYSTEM

Payload No.	028	029
Vehicle	NIKE-NIKE	NIKE-NIKE
CH ₃ OH/H ₂ O Aft Tank		
Wt. Chem. (lbs.)	118	118
Orifice (in.)	0.335	0.335
TiCl ₄ Fwd. Tank		
Wt. Chem (lbs.)	135	135
Orifice (in.)	0.303	0.303
Nose Cone		
Pressure (PSIA)	100	100
Total Payload Wt. (lbs.)	549	548
CG, in. from Nike Joint	24	23½
Launch Date	May 20, 1978	May 22, 1978

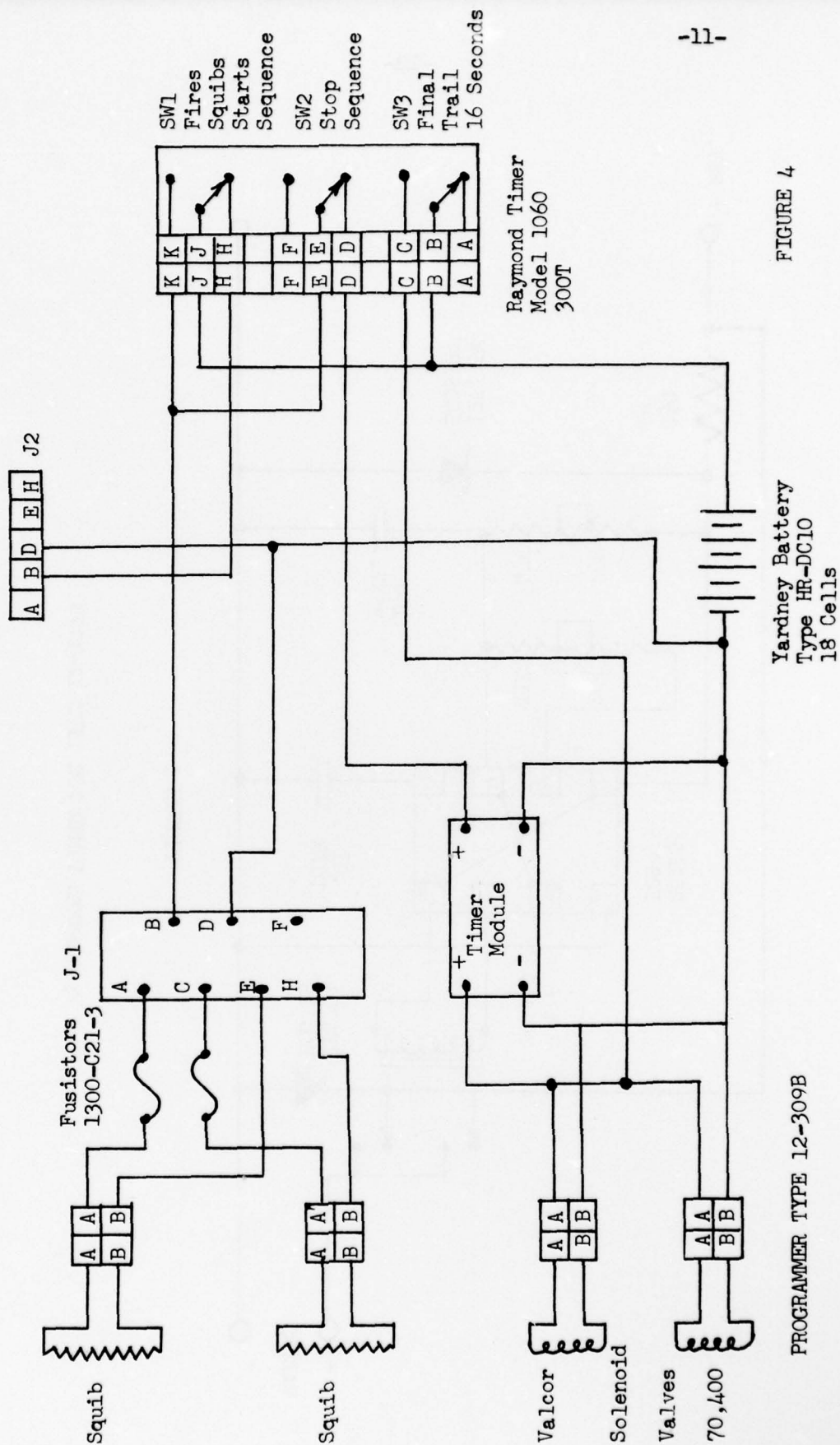


FIGURE 4

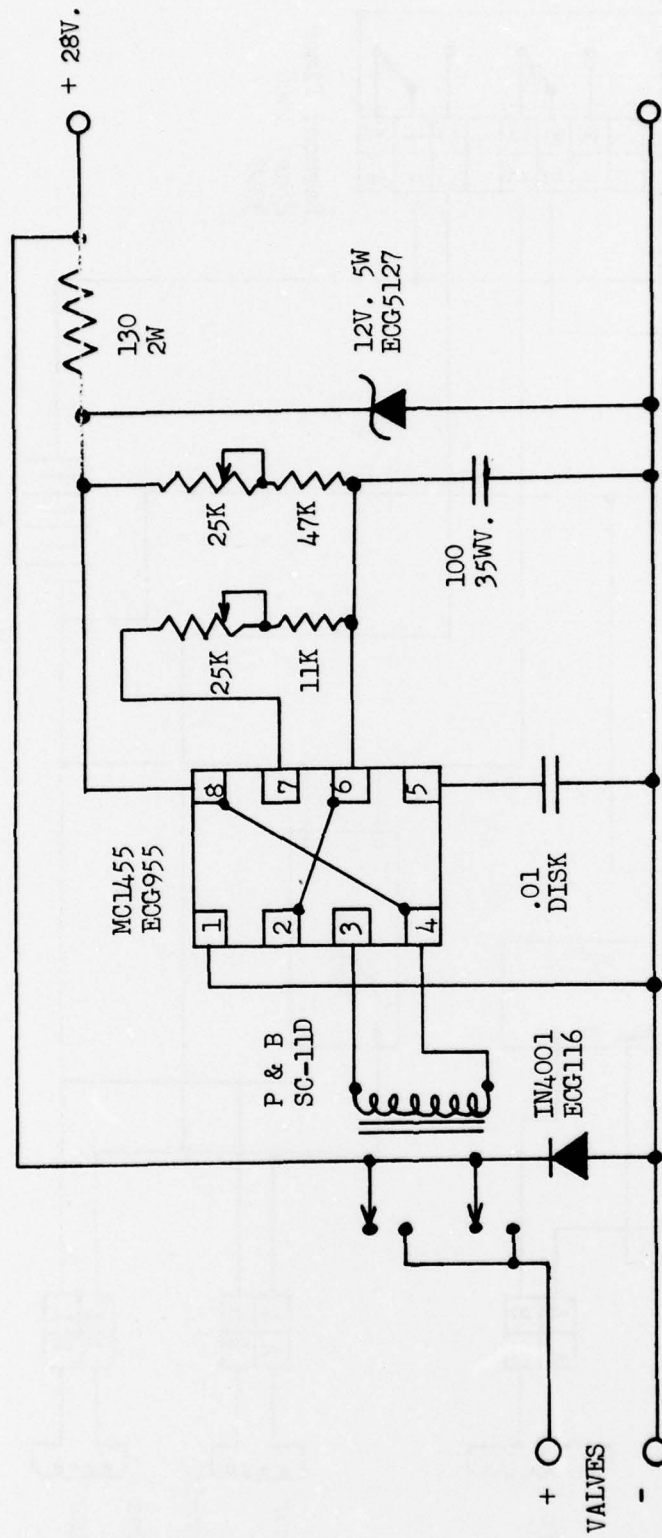


FIGURE 5

PROGRAMMER TIMER FOR TYPE 12-309B

TABLE IV
FORT CHURCHILL
PAYLOAD PARAMETERS, NIKE-NIKE SMOKE SYSTEM MARK III

Payload No.	030	031
Vehicle	NIKE-NIKE	NIKE-NIKE
CH ₃ /H ₂ O Aft Tank		
Wt. Chem (lbs.)	160	161
Orifice (in.)	0.391	0.391
TlCl ₄ Fwd. Tank		
Wt. Chem (lbs.)	184	184
Orifice (in.)	0.348	0.348
Nose Cone		
Pressure PSIA	105	105
Total Payload Wg. (lbs.)	668	668
CG, in. from Nike Joint	28-3/4	28-7/8
Launch Date	Sept. 12, 1978	Sept. 13, 1978

TABLE V
PROGRAMMER TIMING - TRAIL RELEASE
NIKE-NIKE SMOKE SYSTEM
685 lb. - MARK III

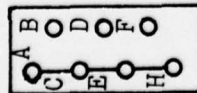
Titanium Tetrachloride Orifice	0.348 in.
Tank Pressure	105 psia
Flow Rate	3.06 lbs./sec.

Methanol/Water Orifice	0.391 in.
Tank Pressure	105 psia
Flow Rate	2.69 lbs./sec.

<u>Trail No.</u>	<u>Start Second</u>	<u>End Second</u>
1	30	36
2	40	46
3	50	56
4	60	66
5	70	76
6	80	86
7	90	96
8	105	120

Programmer Type 12-309A

P-1S
Safe
Plug



P-1A
Arm
Plug

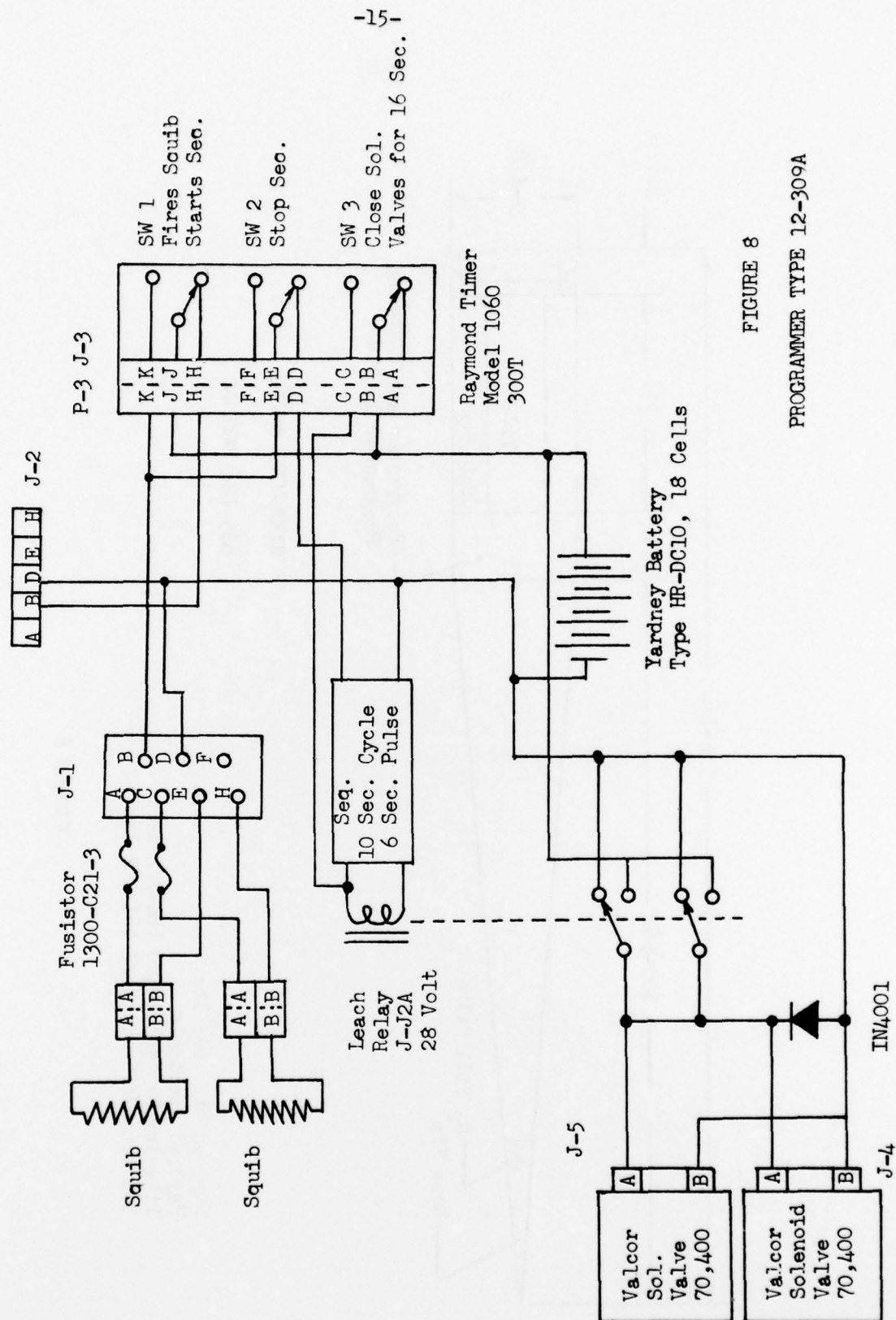
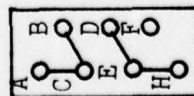
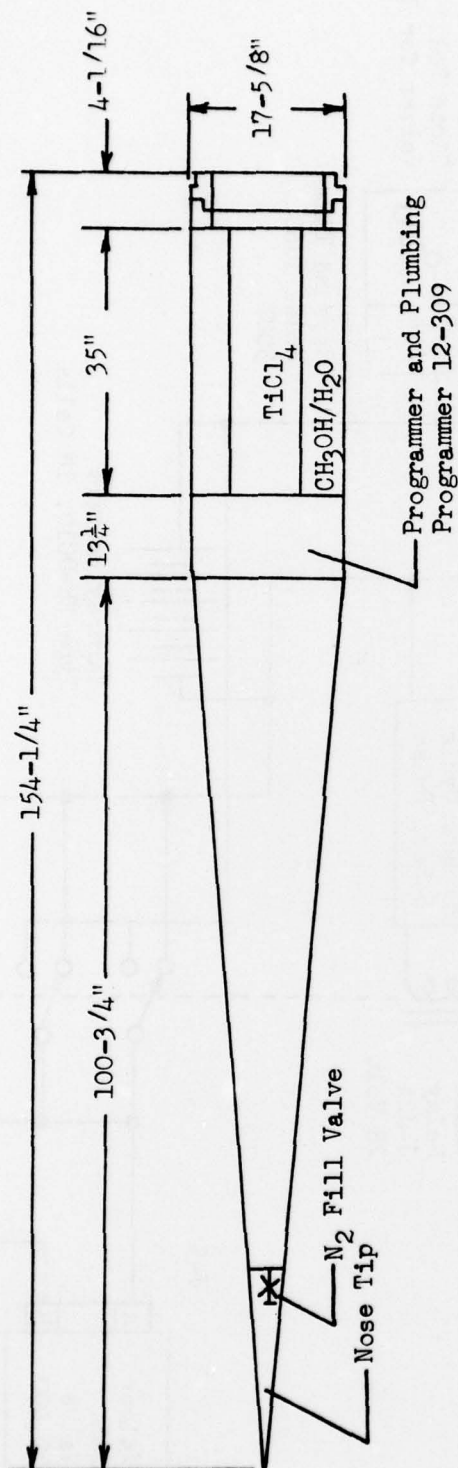


FIGURE 8
PROGRAMMER TYPE 12-309A



NIKE-NIKE SMOKE

685 LB. PAYLOAD

Payload Wt. 685 lbs.
 Payload Chemicals
 Titanium Tetrachloride 184-1/2 lbs.
 36% Water/65% Methanol 161-1/2 lbs.

FIGURE 6

OPERATION HARSES

FLOW DIAGRAM

MARK III

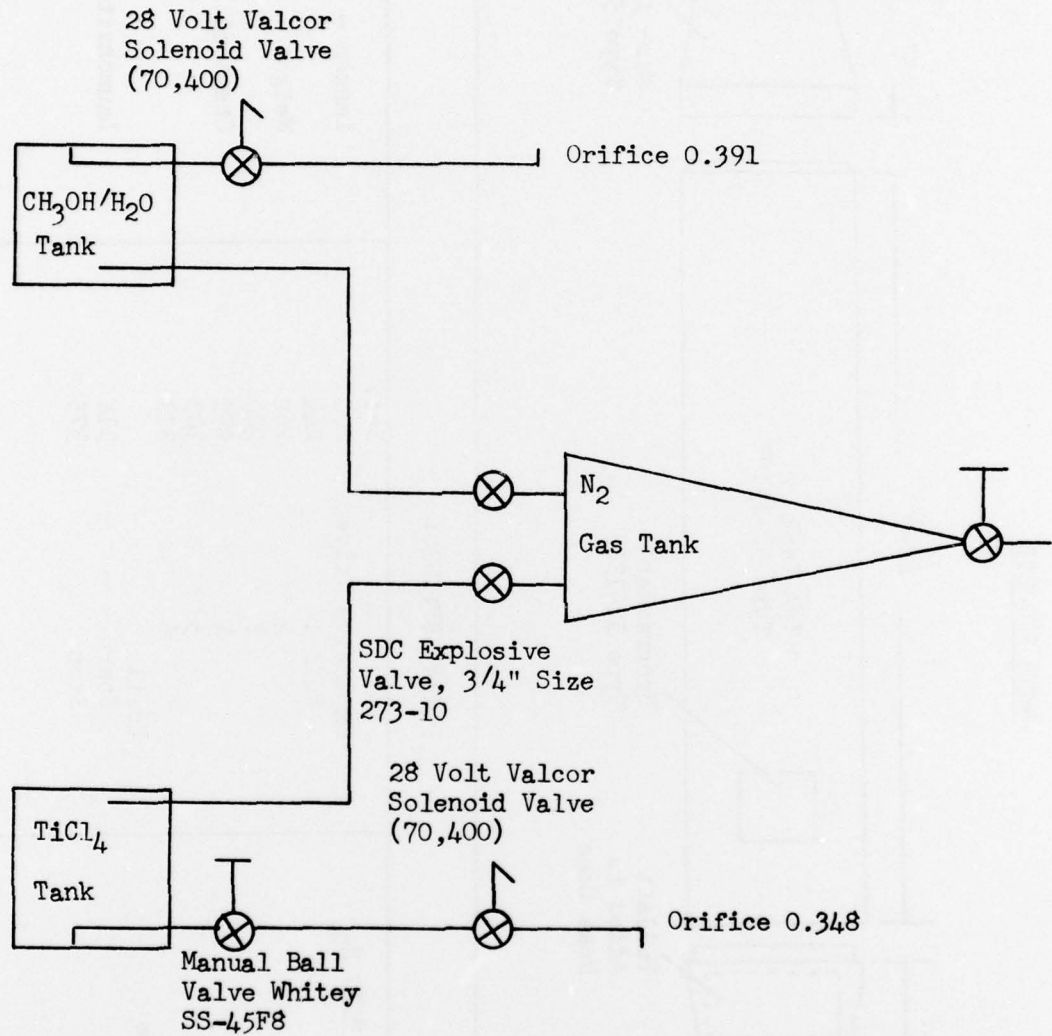
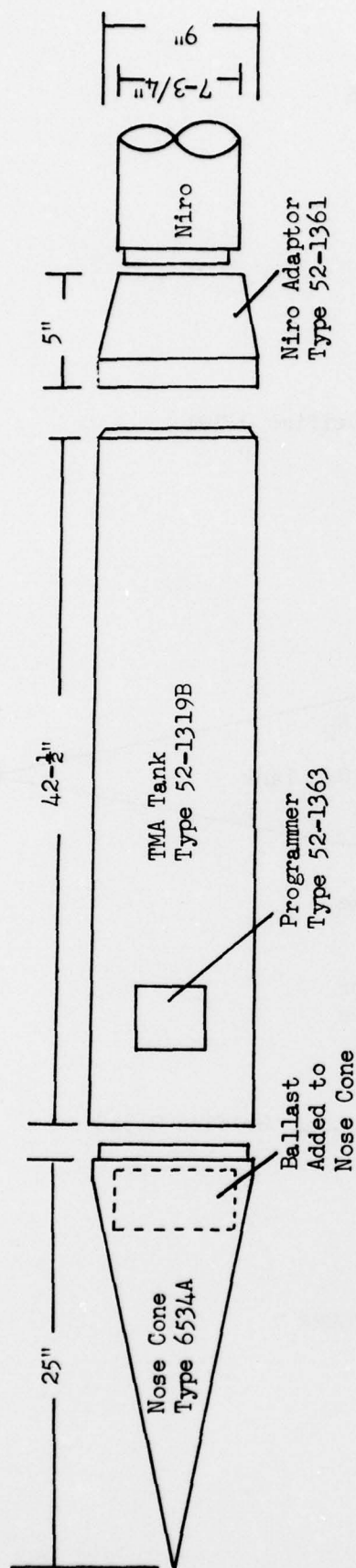


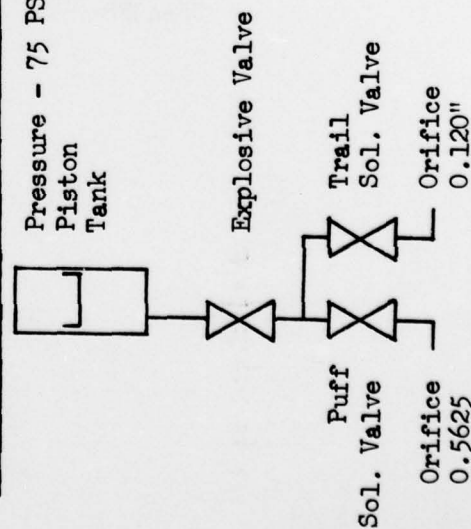
FIGURE 7

DMSP PROJECT



-18-

PLUMBING:



TIMER SETTINGS:

	Sec.
Explosive Valve	200
Puff 1	244
2	262
3	280
4	295
5	303
6	311
Trail	
Start	314
Stop	375

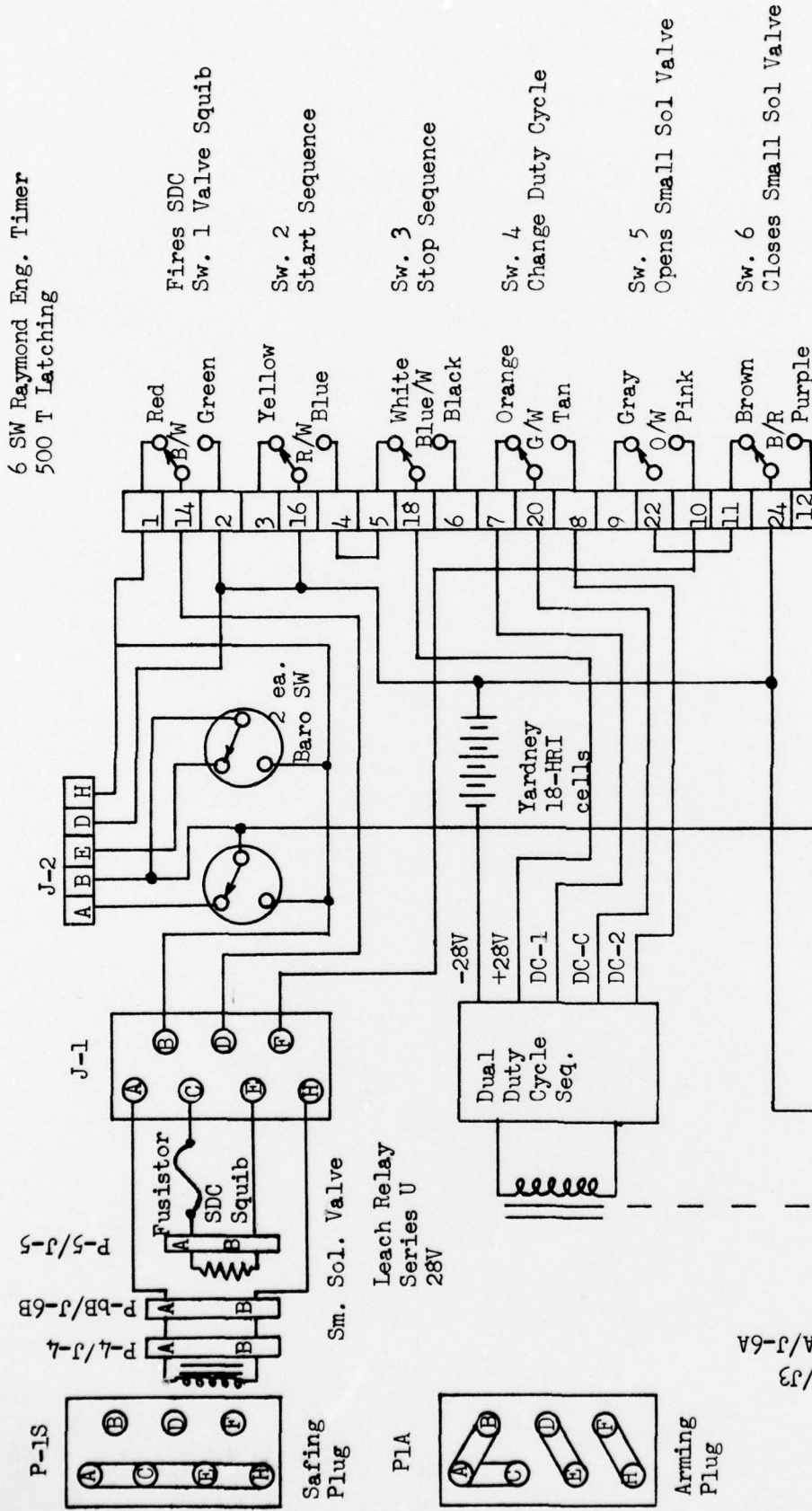
PAYLOAD:

Length - 72.5 inches
 Weight - 130 pounds
 Chemical: Trimethyl Aluminum
 15 pounds
 Launchsite: WSMR Sept. 1977

FIGURE 9

June 30, 1977

6 SW Raymond Eng. Timer
500 T Latching



PROGRAMMER TYPE
52-1363

FIGURE 10

RECEPTACLES		CONNECTORS		PLUGS	
J-1	AMP 126-017	P-1	AMP 126-016		
J-2	AMP 126-011	P-2	Test Lead		
J-3	Part of Valve	P-3	MS3106A-10SL		
J-4	Part of Valve	P-4	MS3106A-10SL		
J-5	PT06P-8-4P	P-5	PT02A-8-4S		
J-6	MS3102A-14S-2S	P-6	MS3106A-14S-2P		

A P P E N D I X

FM--TITANIUM TETRACHLORIDE

Action on metals	Corrosive; no action on steel if FM is dry; vigorous action if FM is moist, FM smoke is corrosive.
Boiling Point	275°F.
Chemical name	Titanium Tetrachloride
Chemical storage group	B
Decomposition temperature	None below boiling point of 275°F.
Decontaminants	Any alkali in solid or solution form
Formula	$TiCl_4$
Freezing Point	Minus 22°F.
Hydrolysis products	Solid $TiOCl$ and HCl ; also $Ti(OH)_4$ if sufficient water is present
Munitions markings	FM SMOKE in black on light green background
Odor	Acrid
Rate of action	Rapid
Rate of hydrolysis	Reacts immediately with water or water vapor
Specific gravity	1.7 at 68°F.
Stability in storage	Stable in steel containers if FM is dry

DESCRIPTION

FM is a liquid compound, titanium tetrachloride, which can be atomized by detonation or by spraying into the air. When it is thus atomized, it hydrolyzes and the smoke soon becomes a composition of solid and liquid particles.

Nitrogen is used with FM to produce pressure. At times a small percentage of other chemicals is added to the compound. The smoke mixture is corrosive and is a colorless to light-yellow liquid that weighs 14 pounds per gallon.

FM reacts vigorously with the moisture in the air to form a dense, white, persistent smoke cloud of finely divided titanium hydrate particles and mist of hydrochloric acid vapor. The formation of the solid particles sometimes clogs spraying apparatus. For this reason it has been replaced to a large extent by FS.

When FM is in a dry state, it has no reaction on steel. If FM is moist, however, it will have a strong corrosive effect on steel.

FM smoke increases in density as the humidity increases. Although a good smoke is produced with average humidity, it tends to dissipate more rapidly than when the humidity is above average.

White FM smoke is considered, under normal conditions, as nontoxic, the liquid burns the skin like a strong acid. The smoke is mildly irritating to the nose and throat at the concentrations found in a smoke cloud, but a protective mask is only required for a heavy concentration.

Detection - heavy colorless liquid having a mildly acrid or pungent odor. Readily detected by large quantity of smoke produced when it leaks from a container.

DECONTAMINATION

Observe the following precautions when handling FM in bulk quantities:

1. Personnel handling FM drums or munitions must wear protective gloves and boots.
2. Personnel handling FM during filling operations, or at other times when this agent could splash on them, should wear goggles, protective aprons or clothing, gloves and boots.
3. It is preferable for personnel operating in an FM smoke cloud to wear protective masks. It is mandatory that masks be worn when the cloud is in a confined space with high concentrations of FM smoke.

4. When FM is spilled, it must be destroyed by repeated dousing with water. Care must be taken to avoid injury from droplets that are likely to be scattered by the violent reaction of FM with water. For this reason, a small amount of water should never be allowed to contact a large amount of FM mixture. If any liquid FM comes into contact with any part of the body, it should be immediately wiped off and the body washed with an abundance of water, then rewashed with a weak solution of bicarbonate of soda or ammonia in water. Contaminated clothing should be removed before washing the body with water or serious burns are likely to result.

STOWAGE

FM is stowed in 55 gallon steel drums. It is stable inside these drums, where it is concentrated, but fumes leaking out past the bung hole plugs will react with moisture in the air to form a corrosive mist that will eat away the outside of the drums.

The drums should be painted on the outside with an acid and weather-proof paint. They should be stowed in well ventilated magazines ashore. (Outdoor stowage is permissible if the outer surfaces of the drums are kept well painted). They should be kept on racks at least 4 to 6 inches off the ground or the floor, as FM vapors are heavy and hug the ground.

Aboard ship the drums should be stowed topside only, but they must be protected from the sun and from salt water spray and constantly inspected for signs of corrosion, leakage and paint deterioration.

The drums should be vented when they have been subjected to direct sunlight or abnormally high temperatures for a protracted period of time. They must also be vented when bulging because of pressure (this calls for extreme caution on the part of personnel doing the venting), or when the drums are to be opened. When

venting is necessary, the drums to be vented should be removed from the place of stowage to prevent contamination of the remaining drums. When it is desired to open FM drums, they should be removed far enough from the place of stowage so the corrosive vapors released when the plug is removed will not be able to contaminate other drums.

When a drum begins to leak badly, the FM should be transferred to any empty nonleaking drum. If no suitable empty drums are available, the leaking drums should be disposed of in order to avoid corroding other drums.

FIRE FIGHTING

If a fire involves or threatens buildings in which FM is stored, all persons within the danger zone shall be notified to vacate until all danger is passed. Fires in magazines shall not be fought. Since a fire involving chemical ammunition is dangerous to the inhabitants of the vicinity, special precautions must be taken to prevent fires in areas where this chemical ammunition is stored. FM is non-flammable, but may cause fires if spilled on flammable material. This is especially true under damp conditions.

36% WATER-64% METHANOL (WM)

Composition	36% H ₂ O-64% Methanol by weight
Specific Gravity at 68°F.	.8834
Freezing Point	Below -50°F.
Stability in Storage	Excellent

The mixture is similar to "anti-freeze" solutions in auto radiators, however the methanol percentage is higher. The mixture is toxic if ingested and fumes can be an irritant if inhaled over a long period. The mixture of water with methanol will reduce the vapor pressure of the methanol and hence the hazard due to inhalation.

Pure methanol is extremely flammable. When mixed with water at these percentages the mixture can be ignited with some difficulty, the alcohol will boil out of solution and burn with the usual blue flame. In general the mixture poses no special handling problems, especially with the small quantities being used (less than 5 gallons).

PHYSICAL PROPERTIES

TRIMETHYLALUMINUM (TMA),

Purified¹

Formula	(CH ₃) ₃ Al
Formula Weight	72.085
State and Color at 25°C (77°F)	Clear, colorless liquid
Stability in Contact with Air	Flames instantly
with Water	Reacts violently
Freezing Point ²	15.3°C (59.5°F)
Boiling Point ² at 760 mm	127.12°C (260.82°F)
Vapor Pressure ^{2,3} at	
20°C (68°F)	9.2 mm
40°C (104°F)	27.2 mm
60°C (140°F)	69.3 mm
80°C (176°F)	157.1 mm
100°C (212°F)	323.3 mm
120°C (248°F)	614.4 mm
140°C (284°F)	1096 mm
Density ⁴ at 25°C (77°F)	0.7478 g/ml
	6.241 lb/gal
Viscosity ⁴ at 25°C (77°F)	1.030 cp
Specific Heat ² at 25°C (77°F)	0.5159 cal/(g)(°C)
	0.5159 btu/(lb)(°F)
Heat of Vaporization at NBP	71 cal/g
	128 btu/lb
ΔH° of Formation ⁵ at 25°C	-27.6 kcal/gram
	formula weight
Heat of Combustion, Net	9,918 cal/g
at 25°C (77°F)	17,840 btu/lb
Heat of Reaction with Water	1,738 cal/g
at 25°C (77°F)	3,127 btu/lb
Coefficient of Volume Expansion	0.001153 per °C
at 25°C (77°F)	0.000641 per °F
Critical Temperature	350°C (662°F)
Critical Pressure	54.2 atm
Soluble in	Hydrocarbons
Freezing Point of 50 wt %	
Solution in TEA	< -76°C (-105°F)
Association Factor in Benzene at 5°C	2.0 (Dimeric)
Thermal Stability	Liquid stable up to 150°C
	(302°F) in inert atmosphere
Heat of Fusion	2.101 kcal/gm mole (monomer)

¹ Sample purity: ≥ 98%.² McCullough and others, J. Phys. Chem. 67, 677 (1963).³ Equation: $\log P \text{ (mm)} = 7.5075 - 1692.6/(t + 237.7)$,
t = °C⁴ Values at other temperatures tabulated on page 61.⁵ Long and Norris, Trans. Roy. Soc. (London) A244, 567
(1959) (recalculated).

TRIETHYLALUMINUM (TEA),

Purified¹

Formula	(C ₂ H ₅) ₃ Al
Formula Weight	114.17
State and Color at 25°C (77°F)	Clear, colorless liquid
Stability in Contact with Air	Flames instantly
with Water	Reacts violently
Freezing Point	-45.5°C (-49.9°F)
	-46.8°C (-52.2°F)
	(two crystalline forms)
Boiling Point at 760 mm	186.6°C (367.9°F)
Vapor Pressure ² at	
60°C (140°F)	0.79 mm
80°C (176°F)	3.5 mm
100°C (212°F)	12.9 mm
120°C (248°F)	40.3 mm
140°C (284°F)	109.8 mm
160°C (320°F)	268 mm
180°C (356°F)	594 mm
200°C (392°F)	1218 mm
220°C (428°F)	2330 mm
Density ³ at 25°C (77°F)	0.8324 g/ml
	6.947 lb/gal
Viscosity ³ at 25°C (77°F)	2.582 cp
Specific Heat at 25°C (77°F)	0.498 cal/(g)(°C)
	0.498 btu/(lb)(°F)
Heat of Vaporization at NBP	120 cal/g
	216 btu/lb
ΔH° of Formation at 25°C	-33.3 kcal/gram
	formula weight
Heat of Combustion, Net	10,202 cal/g
at 25°C (77°F)	18,352 btu/lb
Heat of Reaction with Water	1,109 cal/g
at 25°C (77°F)	1,996 btu/lb
Surface Tension at 28°C (82.4°F)	26.1 dyne/cm
Coefficient of Volume Expansion	0.000815 per °C
at 25°C (77°F)	0.000453 per °F
Dielectric Constant at 25°C	2.58
	(77°F) and 5 megacycles
Critical Temperature	405°C (761°F)
Critical Pressure	134 atm
Soluble in	Hydrocarbons
Freezing Point of 50 wt %	
Solution in TMA	< -76°C (-105°F)
Freezing Point of 50 wt %	
Solution in DEAH	-64°C (-83.2°F)
Association Factor in Benzene at 5°C	2.0 (Dimeric)
Decomposition Rate ⁴ of Liquid	
in Bomb at 140°C (284°F)	0.0028 %/hr
160°C (320°F)	0.023 %/hr
180°C (356°F)	0.16 %/hr
200°C (392°F)	0.90 %/hr
220°C (428°F)	0.075%/min
240°C (464°F)	0.33 %/min
260°C (500°F)	1.3 %/min

¹ Sample purity: 99 wt % except for the following properties for which it was 95%: density, viscosity, coefficient of volume expansion, dielectric constant, and decomposition rate.² Equation: $\log P \text{ (mm)} = 9.0086 - 2369.1/(t + 200)$,
t = °C

Experimental range: 80-190°C.

³ Values at other temperatures tabulated on page 62.⁴ Calculated assuming one mole gas evolved per gram formula weight TEA decomposed. Experimental range: 160-270°C.Equation: $\log (\%/hr) = 17.249 - 8185/T$, T = °K